Claims

[c1] 1. A pressure-sealing, pressure-monitoring closure for non-invasively sealing a reaction vessel to a defined release pressure in microwave-assisted chemistry, said closure comprising:
a pressure-resistant, microwave-transparent reaction vessel, one portion of which defines a mouth for said vessel;
a flexible pressure-transmitting releasable cover assembly on said mouth of said vessel;
a pressure transducer on said cover assembly and external to said vessel for monitoring the pressure in said vessel as exerted against said flexible cover assembly; and
a clamp for urging said vessel, said cover assembly and said transducer together under a defined force so that when the pressure in said vessel exceeds the defined force, said cover assembly can flex and release the pressure from said vessel.

[c2] 2. A closure according to Claim 1 and further comprising:
a microwave source for applying microwave radiation to said vessel and its contents; means for moderating the microwave radiation applied to the vessel; and
a processor in signal communication with said source, said moderating means and said transducer for moderating the microwaves applied by said source in response to the pressure in said vessel as measured by said transducer.

[c3] 3. A closure according to Claim 2 wherein said microwave moderating means comprises means for changing the power of said microwave source.

[c4] 4. A closure according to Claim 2 wherein said microwave moderating means comprises means for changing the duty cycle of the source.

[c5] 5. A closure according to Claim 2 wherein said microwave moderating means comprises means for optically adjusting the microwaves applied to the source.

[c6] 6. A closure according to Claim 1 wherein said clamp includes means for adjusting the force with which the clamp urges said vessel, said transducer and

said cover together to thereby define the pressure at which said cover can flex and release pressure from said vessel.

[c7] 7. A closure according to Claim 6 wherein said adjusting means comprises:
a motor; and
a ram in force-transmitting relationship with said transducer and incrementally driven by said motor for adjustably and incrementally changing the force with which said transducer is clamped to said cover.

[c8] 8. A closure according to Claim 1 and wherein said cover assembly comprises a flexible septum and a flexible cap for maintaining said septum on said mouth of said vessel in a gas-tight relationship independent of said clamp and without interfering with said clamp when said clamp urges said vessel, said cover assembly and said transducer together.

[c9] 9. A closure according to Claim 8 wherein said cap fixes the perimeter of said septum to the perimeter of said mouth while leaving said transducer in contact with said septum.

[c10] 10. A closure according to Claim 1 wherein said transducer includes a transducer button immediately adjacent said cover assembly for insulating said transducer from excess heat generated by a reaction in said vessel and for protecting said transducer from direct chemical contact with the contents of said vessel if said cover assembly flexes sufficiently to open said vessel.

[c11] 11. A closure according to Claim 1 wherein said reaction vessel is formed of a material selected from the group consisting of glass, quartz and polymers.

[c12] 12. A closure according to Claim 8 wherein said septum is selected from the group consisting of rubbers and polymers and said flexible cap is polymeric.

[c13] 13. A closure according to Claim 12 wherein said septum is selected from the group consisting of butyl rubbers and siloxane polymers and said cap comprises polypropylene.

[c14] 14. A method for non-invasively monitoring and releasing pressure in a reaction vessel in microwave-assisted chemistry, the method comprising:

clamping a microwave-transparent reaction vessel and a pressure-sensing transducer together with the transducer and the vessel cover in pressure-transmitting contact with one another;
taring the clamping force from the transducer"s measurement so that the transducer measures the net force exerted by pressure in the vessel and against the cover;
applying microwave radiation to the vessel and its contents; and
monitoring the pressure sensed by the transducer as the microwaves are applied to the vessel;
characterized in that:
the clamping step comprises clamping a flexible cover assembly to the vessel and adjusting the clamping force to a predetermined applied amount so that when pressure in the vessel exceeds the predetermined applied amount, the cover will flex and release the excessive pressure.

[c15] 15. A method of microwave-assisted chemistry according to Claim 14 comprising the steps of:
adding reactants to the vessel;
placing the cover on the vessel; and
sealing the cover to the vessel with a gas-tight seal;
all prior to the step of clamping the vessel, the cover and the transducer together.

[c16] 16. A method of microwave-assisted chemistry according to Claim 14 comprising the steps of:
stopping the applied microwaves;
allowing the vessel to cool; and
unclamping the vessel to release any residual pressure.

[c17] 17. A method of microwave-assisted chemistry according to Claim 14 and further comprising moderating the microwave radiation applied to the vessel in response to the measured pressure.

[c18] 18. A method of microwave-assisted chemistry according to Claim 17 wherein the step of moderating the microwave radiation comprises programming a

processor that is in signal communication with the microwave source applying the radiation and in signal communication with the transducer.

[c19] 19. A method of microwave-assisted chemistry according to Claim 14 wherein the step of taring the clamping force comprises:
recording the initial clamping force as measured by the transducer prior to applying the microwave radiation; and
ignoring the initial force when measuring the pressure with the transducer as the microwaves are being applied.

[c20] 20. A method of microwave-assisted chemistry according to Claim 14 wherein the step of taring the clamping force comprises programming the transducer to ignore the initial clamping force prior to the step of applying the microwave radiation.

[c21] 21. A method of microwave-assisted chemistry according to Claim 20 wherein the step of programming the transducer comprises programming a processor that is in signal communication with said transducer.

[c22] 22. A method of noninvasive pressure measurement and control in microwave assisted chemistry, the method comprising:
urging a transducer against a flexible, pressure-releasing portion of a microwave-transparent reaction vessel;
using the transducer to measure the initial force with which the transducer is urged against the vessel;
applying microwave radiation to the vessel and its contents to initiate or promote a chemical reaction therein;
monitoring any increased force exerted by the vessel against the transducer as the chemical reaction proceeds;
moderating the applied microwave radiation based upon the difference between the initial urging force and the increased force; and
releasing any excess pressure through the pressure-releasing portion.

[c23] 23. A pressure measurement method according to Claim 22 wherein the step of urging the transducer against the vessel comprises urging the transducer in

contact against a flexible cover assembly on the mouth of the vessel.

[c24] 24. A pressure measurement method according to Claim 22 wherein the step of measuring the initial force comprises sending a signal from the transducer to a processor in communication with the transducer and from the processor to an output in communication with the processor.

[c25] 25. A pressure measurement method according to Claim 22 wherein the step of applying microwave radiation comprises applying the radiation from a source of microwave radiation.

[c26] 26. A pressure measurement method according to Claim 22 wherein the step of monitoring the increased force comprises forwarding a signal from the transducer to a processor in communication with the transducer.

[c27] 27. A pressure measurement method according to Claim 26 and further comprising forwarding a pressure-measurement signal from the processor to an output.

[c28] 28. A pressure measurement method according to Claim 22 wherein the step of moderating the microwave radiation comprises moderating the microwave power.

[c29] 29. A pressure measurement method according to Claim 22 wherein the step of moderating the microwave radiation comprises moderating the microwave duty cycle.

[c30] 30. A pressure measurement method according to Claim 22 wherein the step of moderating the microwave radiation comprises optically moderating the microwave radiation.

[c31] 31. An instrument for microwave-assisted chemistry comprising:
a source of microwave radiation;
a cavity in wave communication with said source;
a vessel holder associated with said cavity for holding a reaction vessel in said cavity for exposure to microwaves from said source;
a flexible, pressure-releasing cover assembly for a reaction vessel;

a vessel clamp for engaging portions of said cavity, said cover assembly and a reaction vessel when a vessel is in said vessel holder; and
a transducer in said clamp for bearing against a vessel in said vessel holder when said clamp engages said cavity with a vessel therein.

[c32] 32. An instrument according to Claim 31 comprising means for adjusting the transducer so that said transducer measures net pressure from the vessel rather than gross pressure applied by said vessel clamp.

[c33] 33. An instrument according to Claim 32 wherein said transducer adjusting means comprises:
a processor in signal communication with said transducer;
an input in communication with said processor; and
an output in signal communication with said processor.

[c34] 34. An instrument according to Claim 31 wherein said vessel holder includes a microwave attenuator.

[c35] 35. An instrument according to Claim 34 wherein said clamp engages said attenuator.

[c36] 36. An instrument according to Claim 34 or 35 wherein said clamp engages aid vessel in said attenuator.

[c37] 37. An instrument according to Claim 32 and further comprising a processor in signal communication with said source and said transducer for moderating the microwaves from said source in response to pressure measurements from said transducer.

[c38] 38. An instrument according to Claim 31 wherein said microwave source is selected from the group consisting of magnetrons, klystrons and solid state sources.

[c39] 39. An instrument according to Claim 31 wherein said cavity comprises a single mode cavity.

[c40] 40. An instrument according to Claim 31 and further comprising a waveguide

between said source and said cavity for directing microwaves from said source to said cavity.

[c41] 41. An instrument according to Claim 31 and further comprising means for measuring the temperature in said cavity.

[c42] 42. An instrument according to Claim 41 wherein said temperature measuring means is in signal communication with said processor for moderating the application of microwaves from said source in response to the measured temperature.

[c43] 43. An instrument according to Claim 37 and further comprising means for adjusting the force with which said clamp engages said transducer against a vessel.

[c44] 44. An instrument according to Claim 43 and further comprising input and output means to and from said processor for programming said processor to apply a predetermined engaging force to said clamp and said transducer against a vessel.

[c45] 45. A vessel for microwave chemistry comprising:
a microwave-transparent well for holding reactants;
a mouth in communication with said well;
a flexible septum on said mouth; and
a flexible cap for engaging said mouth and for securing said flexible septum in a pressure-sealing relationship on said mouth that is defined by the flexing strength of said cap to thereby maintain said septum in the pressure sealing relationship under pressures less than the flexing strength of said cap and for permitting said cap and said septum to flex and controllably release pressures in said vessel that are greater than the flexing strength of said cap.

[c46] 46. A vessel according to Claim 45 wherein said septum and said cap comprise a ingle integral piece.

[c47] 47. A vessel according to Claim 45 wherein said septum and said cap comprise separate pieces.

[c48] 48. A vessel according to Claim 47 wherein said cap comprises polypropylene and said septum is selected from the group consisting of butyl rubber and siloxane polymers.

[c49] 49. A vessel according to Claim 45 wherein said cap has a flexing strength of at least one atmosphere.

[c50] 50. A vessel according to Claim 45 wherein said cap has a flexing strength of at least two atmospheres.